Radiation hardness of wax

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https://indico.cern.ch/event/1599118/

Radiation hardness requirements of impregnation resins for FCC-hh superconducting magnets – training quenches

Final focus magnets

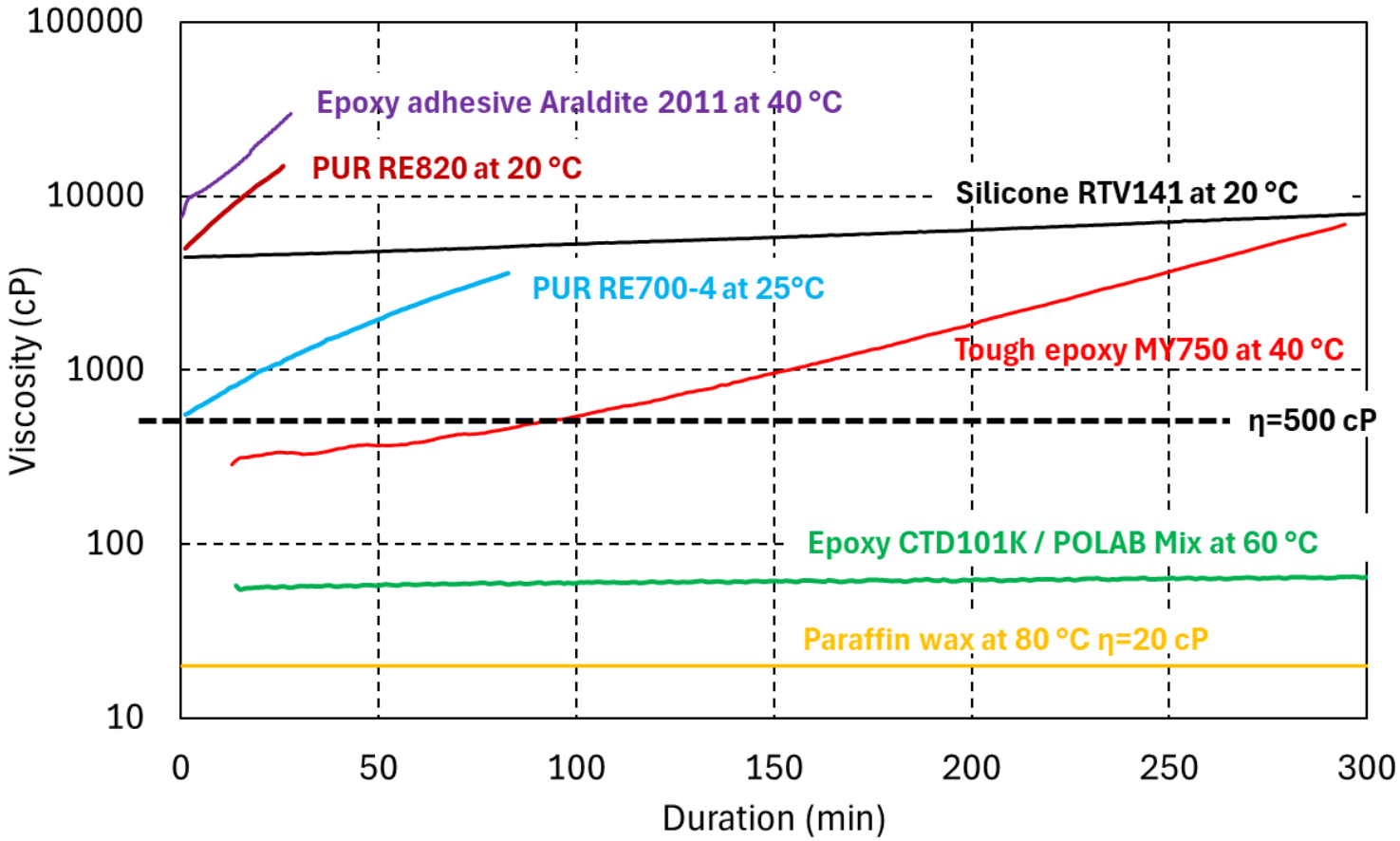
- Choice of the impregnation resin is dictated by high radiation doses.
- The higher the dose that the organic materials resist, the less shielding is needed and the better the performance of the magnet.
- 4 cm-thick tungsten shielding is needed to limit the irradiation dose to 80 MGy [1],[2].
- The time needed to train few individual final focus magnets to the target current is comparatively small.

Main magnets in the arcs

- Resins that are not extremely radiation hard can be considered, for instance:
  - Anhydride free (tough) epoxy systems
  - Wax
- Thousands of magnets need to be tested. The number of training quenches to reach the target current of the individual virgin magnets should be as low as possible.

[1] Future Circular Collider Study. Volume 3: The Hadron Collider (FCC-hh) Conceptual Design Report, preprint edited by M. Benedikt et al. Eur. Phys. J. Spec. Top. 228 (2019) 755-1107.
[2] B. Humann, "Energy Deposition Studies for the Experimental Insertions of FCC-hh", Diplomarbeit, TU Wien, (2019)

Choice of impregnation resins: processibility



Viscosity as a function of duration at the processing temperature.
Courtesy C. Urscheler and M. Albeck.

Wax types, irradiation and sample characterisation

- Ten different wax types including pure natural and synthetic waxes, wax mixtures and wax composites, have been exposed to ^{60}Co gamma irradiation in ambient air with an approximate dose rate of 2 kGy/h.

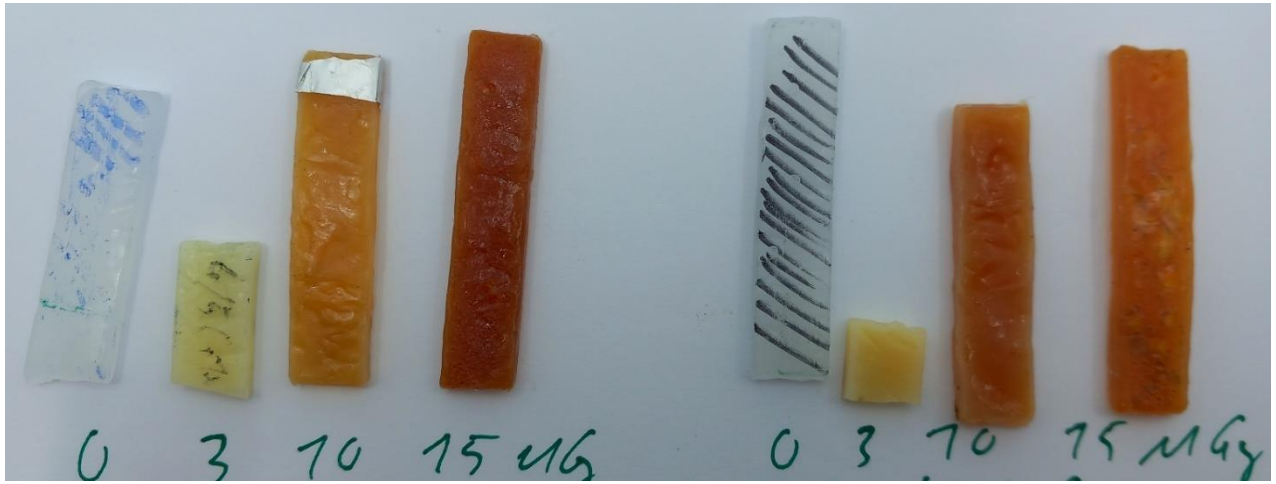
1. Polarit A55, high-purity paraffin wax hydrocarbon chain lengths ranging from C20 to C50 (0, 1.6, 3, 10, 15 MGy)
2. Polarit A55 with S2-glass [0/90] (0, 3, 10, 15 MGy) [3]
3. Bees wax (0, 1.6, 3, 10, 15 MGy)
4. TeCe Ozokerit Z130 mineral wax (0, 1.6, 3, 10 MGy)
5. Polarit 54/56-Carnauba wax-45vol.%Al_2O_3* [4] (0, 1.6, 3, 5, 10, 15, 25, 30 MGy)
6. Polarit 54/56+Irganox1010+Irga 168 *(0, 3, 10, 15 MGy)
7. Polarit 54/56 + UV stabilizer 1% *(0, 3, 10, 15 MGy)
8. T19/45PB4/ 45vol.%Al_2O_3 *(0, 3, 10 MGy)
9. HA wax / 45vol.%Al_2O_3 *(0, 3, 10 MGy)
10. HB wax / 45vol.%Al_2O_3 *(0, 3, 10, 15 MGy)

- * Wax samples have been received from A. Brem, PSI.
- Assessment of radiation damage is based on:
  - visual aspect
  - melting enthalpy and melting temperature as revealed by Differential Scanning Calorimetry (DSC)
  - 3-point bending tests of composites

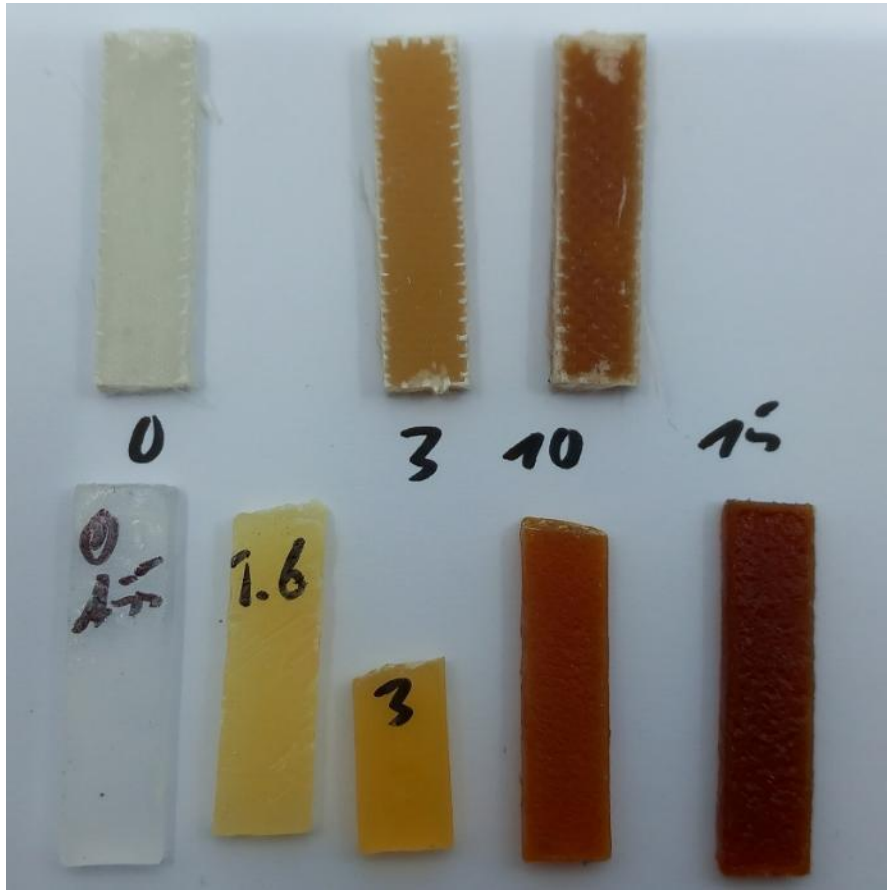
[3] J. Bertsch, C. Scheuerlein, P. Wiker, D.M. Parragh, A. Echtermeyer, R. Piccin, "*Thermomechanical properties of epoxy and wax matrix composites for superconducting magnets*", 2025 *IOP Conf. Ser.: Mater. Sci. Eng.* **1327** 012204

[4] A. Brem et al, "*From Hot to Cold: Advanced Materials and Processes for Nb3Sn Based Magnets*", IEEE Trans. Appl. Supercond., 34(5), (2024), Art. No. 7700105, https://ieeexplore.ieee.org/stamp/stamp.jsp?tp=&arnumber=10339837 .

Radiation effect on visual aspect of unfilled paraffin wax



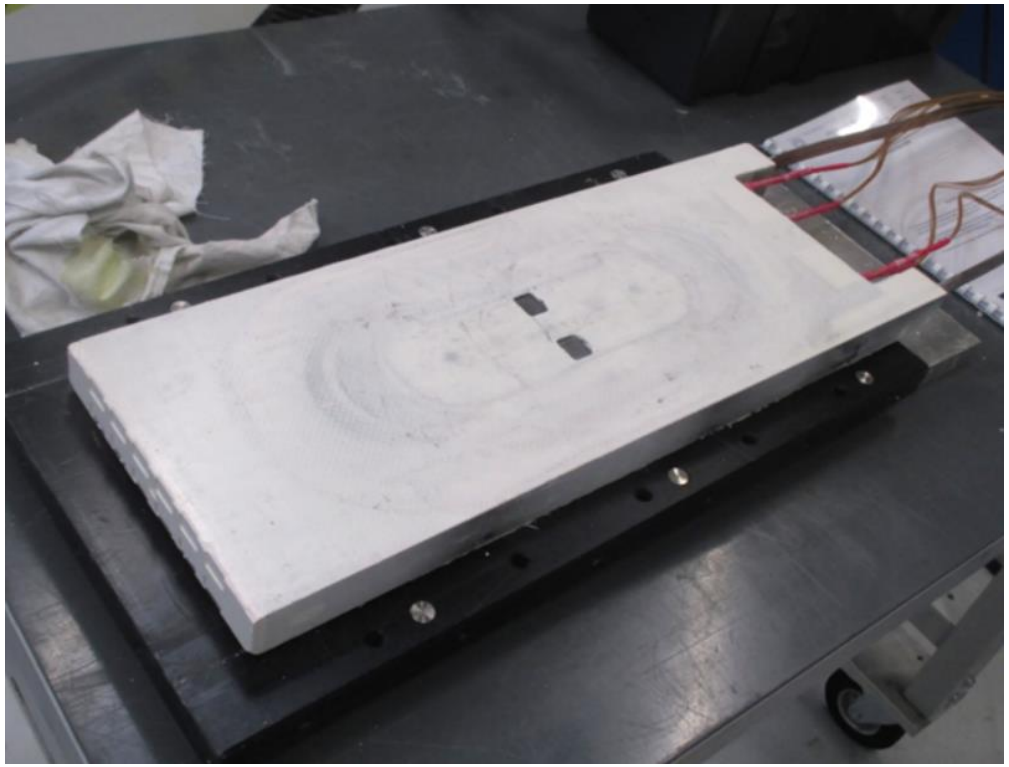
Paraffin wax Polarit 54/56 with (a) UV stabiliser and (b) antioxidants before and after irradiation to 3 MGy, 10 MGy and 15 MGy.



Paraffin wax Polarit A55-S2 glass composite (top) and pure Polarit A55 before and after irradiation up to 15 MGy.

Al_2O_3 filled wax for Nb_3Sn coil impregnation

- Polarit 54/56-Carnauba wax-45vol.%Al_2O_3 mixture (in the following referred to as “PSI standard”) is a PSI development that is commonly used for the impregnation of stress managed coils.
- Recently at CERN a SMC coil has been impregnated with the PSI standard wax mixture to test for the first time if this impregnation system can be used in coils where a pre-stress needs to be applied.

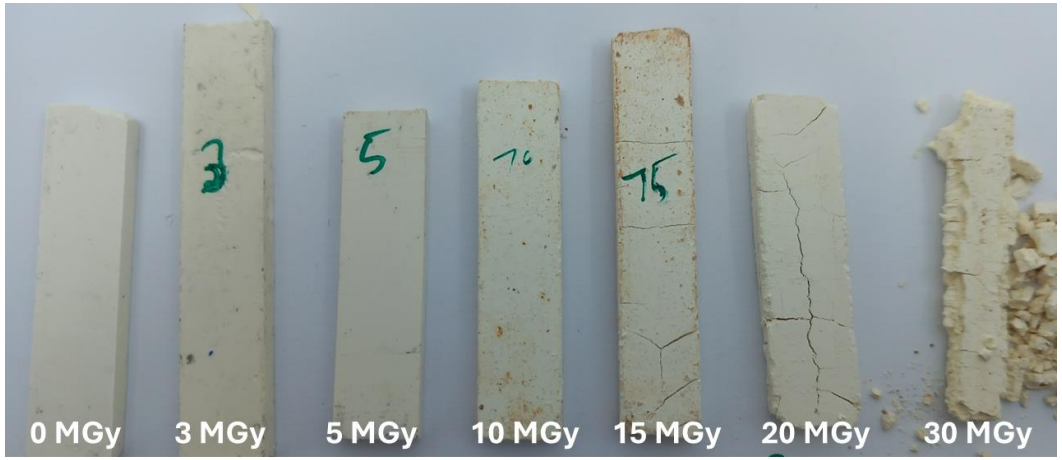


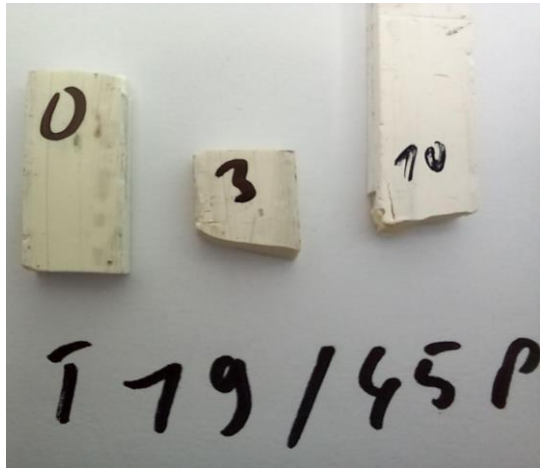
SMC11T_2G coil impregnated with Polarit 54/56 / Carnauba wax / 45vol.%Al_2O_3 mixture [5].

[5] From A. Haziot, “Assembly of SMC with wax”, presentation at TE-MSC-SMT section meeting 28. October 2025, https://indico.cern.ch/event/1595969/

Radiation effect on visual aspect of Al_2O_3 filled samples

- PSI standard wax mixture samples get first cracks after 10 MGy.
- Visual aspect of HA, HB and T19/45PB4 samples is unchanged up to at least 10 MGy (the white sample colour comes from Al_2O_3, which is not modified by gamma irradiation).

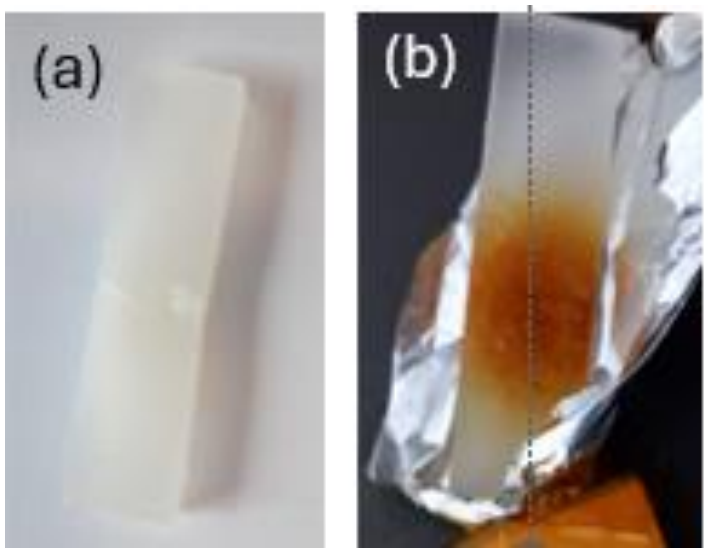




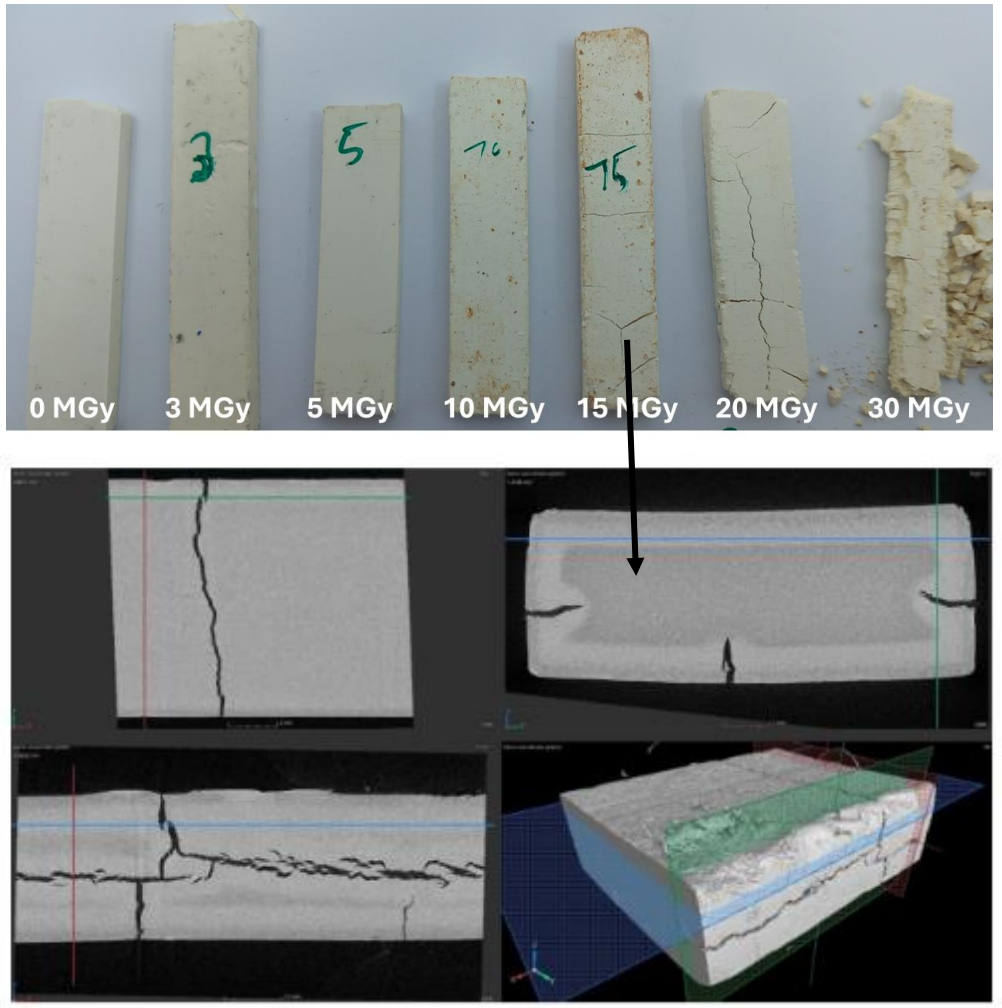
Photographs of (a) PSI standard mixture, (b) T19/45PB4- 45vol.%Al_2O_3 and (c) HA- 45vol.%Al_2O_3 and HB 45vol.%Al_2O_3.

Effect of irradiation environment

- Strong radio oxidation during irradiation in ambient air is visible in tomographic cross sections of PSI standard wax samples.
- Different visual aspect of paraffin wax after 10 MGy in liquid helium and in ambient air.



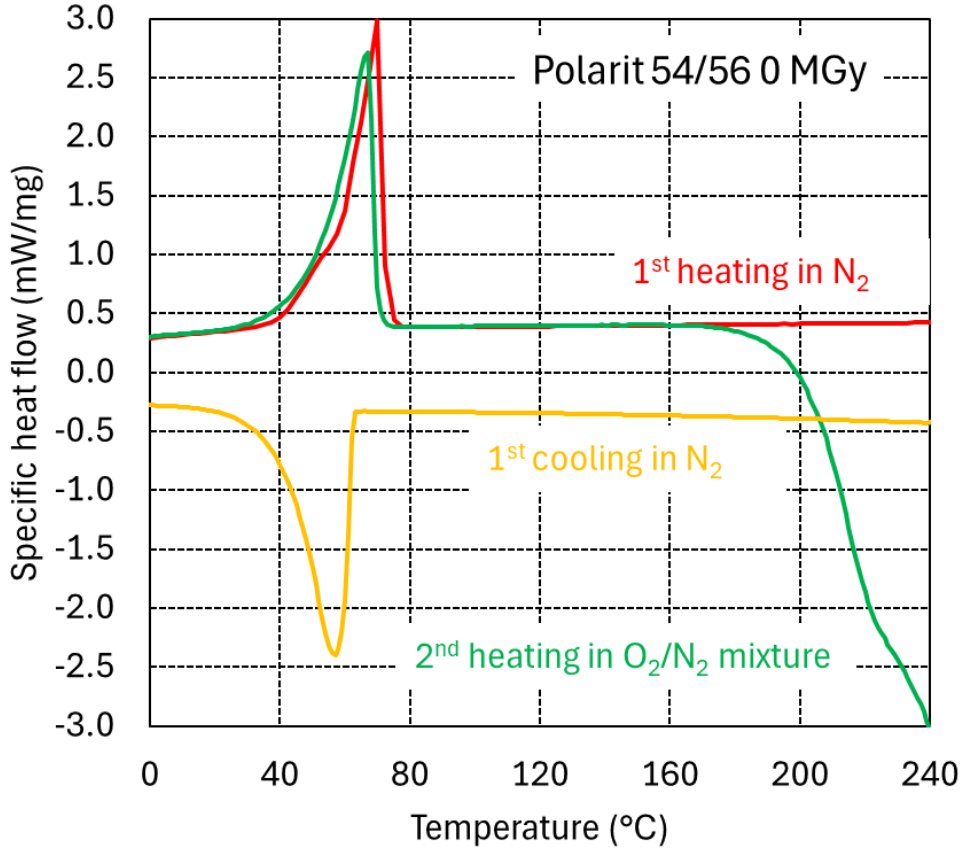
Paraffin wax Polarit A55 after 10 MGy irradiation with a 24 GeV/c proton beam in (a) liquid helium and (b) ambient air.



µ-CT images of PSI standard wax mixture after 15 MGy absorbed in ambient air.

Differential Scanning Calorimetry (DSC)

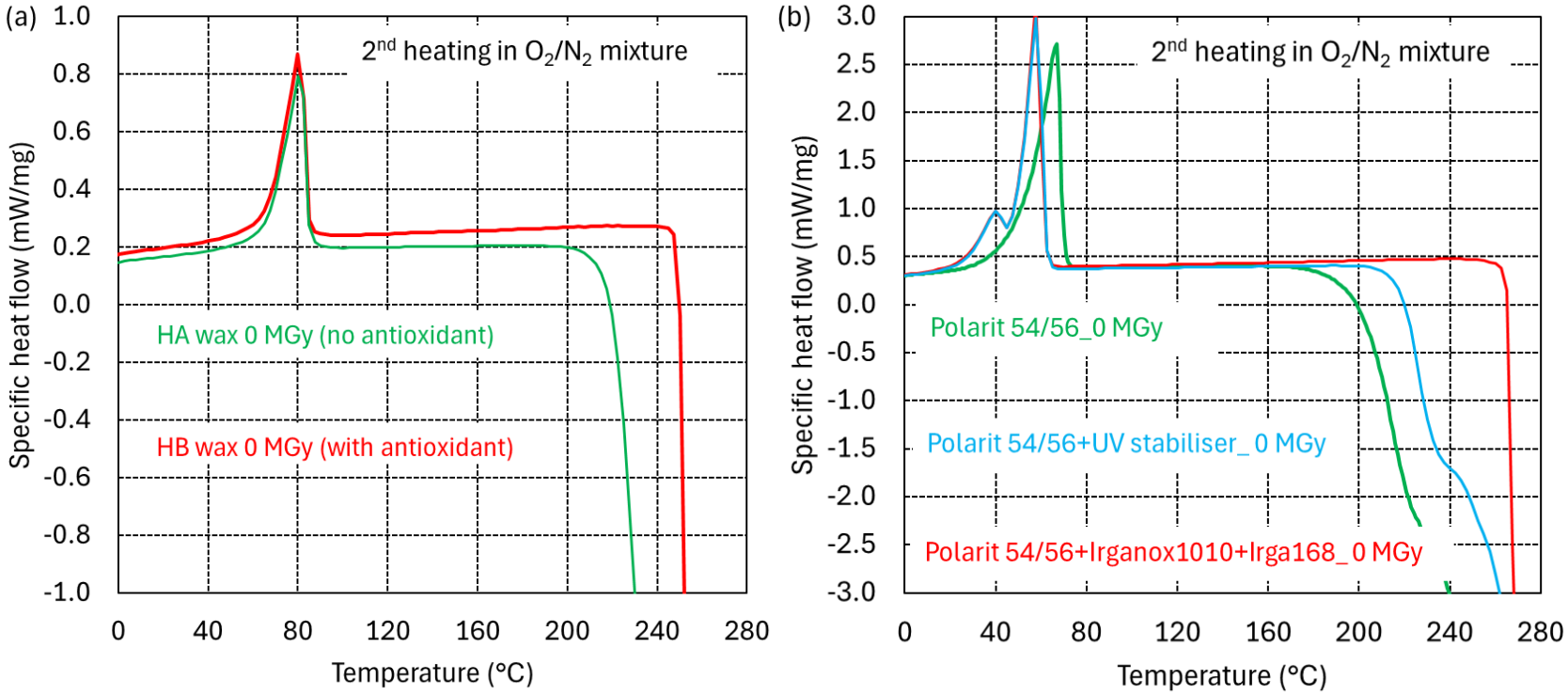
- Measures the change of the heat flux through the sample during a heat ramp of 10 K/minute.
  - 1st heating in inert gas
  - 1st cooling in inert gas
  - 2nd heating to >250 °C in oxygen containing gas
- Endothermic heat flows (melting enthalpies) are presented as positive values and exothermic heat flows (crystallisation enthalpies) as negative values.
- Dose dependent changes of the melting temperature, melting enthalpy, and the onset of degradation in oxygen containing atmosphere are monitored.



Specific heat flow through Paraffin wax Polarit A54/56 during thermal cycle in inert gas (1st heating and colling) and in O_2/N_2 gas mixture (2nd heating cycle).

Effect of antioxidants on degradation temperature

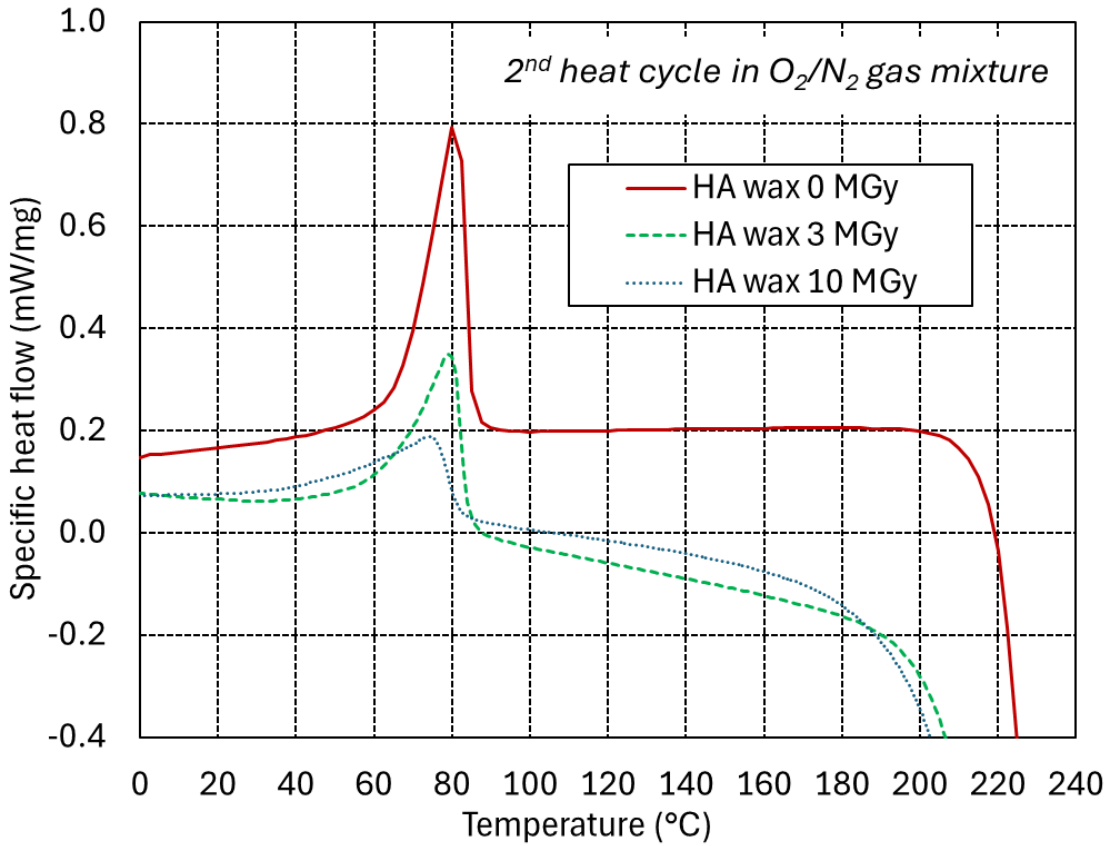
- Addition of UV stabilizer or antioxidant increases the degradation onset temperature.
  - Polarit 54/56: 202 °C
  - Polarit 54/56+Irganox 1010+Irga 168: 265 °C
  - Polarit 54/56+UV stabilizer: 218 °C
- HA and HB wax degradation onset temperatures are 220 °C and 250 °C, respectively.
- Small effect on melting temperature and melting enthalpy.



Specific heat flow through (a) HA wax with and without antioxidant, and (b) paraffin wax Polarit A54/56 without and with UV stabiliser or antioxidants.

Effect of irradiation dose on melting enthalpy, melting temperature and degradation temperature

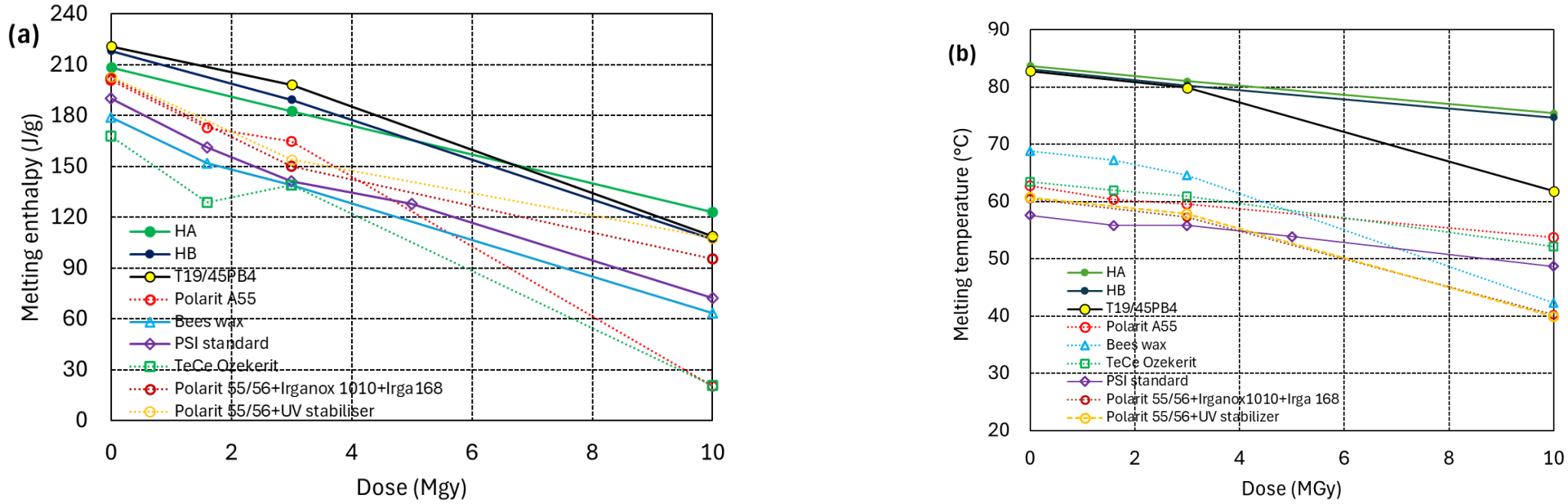
- Melting enthalpy decreases with increasing dose, indicating irradiation induced chain scission.
  - HA_0 MGy: 208 J/g
  - HA_3 MGy: 183 J/g
  - HA_10 MGy: 123 J/g
- Melting temperature (defined as specific heat peak maximum) decreases.
  - HA_0 MGy: 84 °C
  - HA_3 MGy: 81 °C
  - HA_10 MGy: 75 °C
- Degradation onset temperature decreases with increasing dose.



Specific heat flow through "HA" wax before and after irradiation.

Melting enthalpy and melting temperature as a function of dose

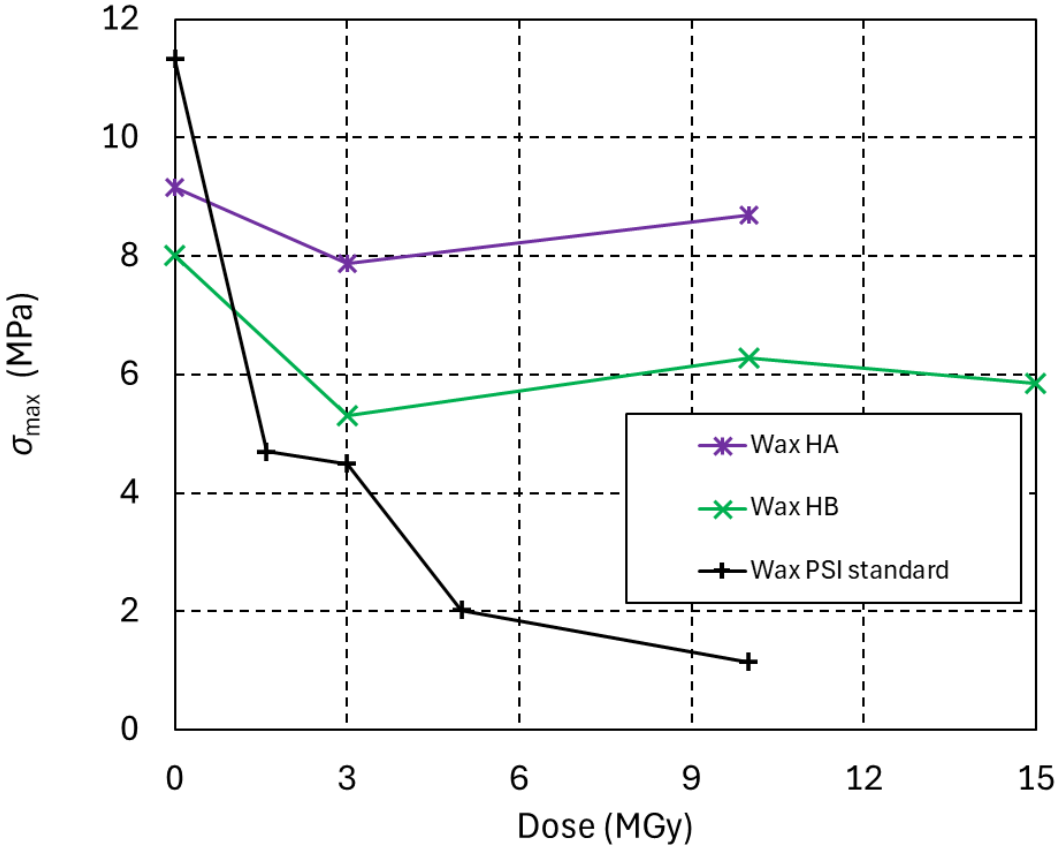
- Similar trend is observed for all wax types. Radiation induced chain scission reduces melting enthalpy and melting temperature with increasing dose.
- In average, after 3 MGy the melting enthalpy is reduced by 19% and the melting temperature is reduced by 3 °C.
- Some small differences between the different wax types might be explainable by the limited number of tests (analysis is based on 1 DSC measurement per wax type and dose step).



(a) Melting enthalpy and (b) melting temperature of different wax types as a function of dose. Al_2O_3 filled samples normalised to wax mass, assuming 77.5% of sample mass is from Al_2O_3. Melting temperature is defined as the temperature at the specific heat flow maximum during the 1st heating cycle.

Radiation damage monitored by 3-point bending tests

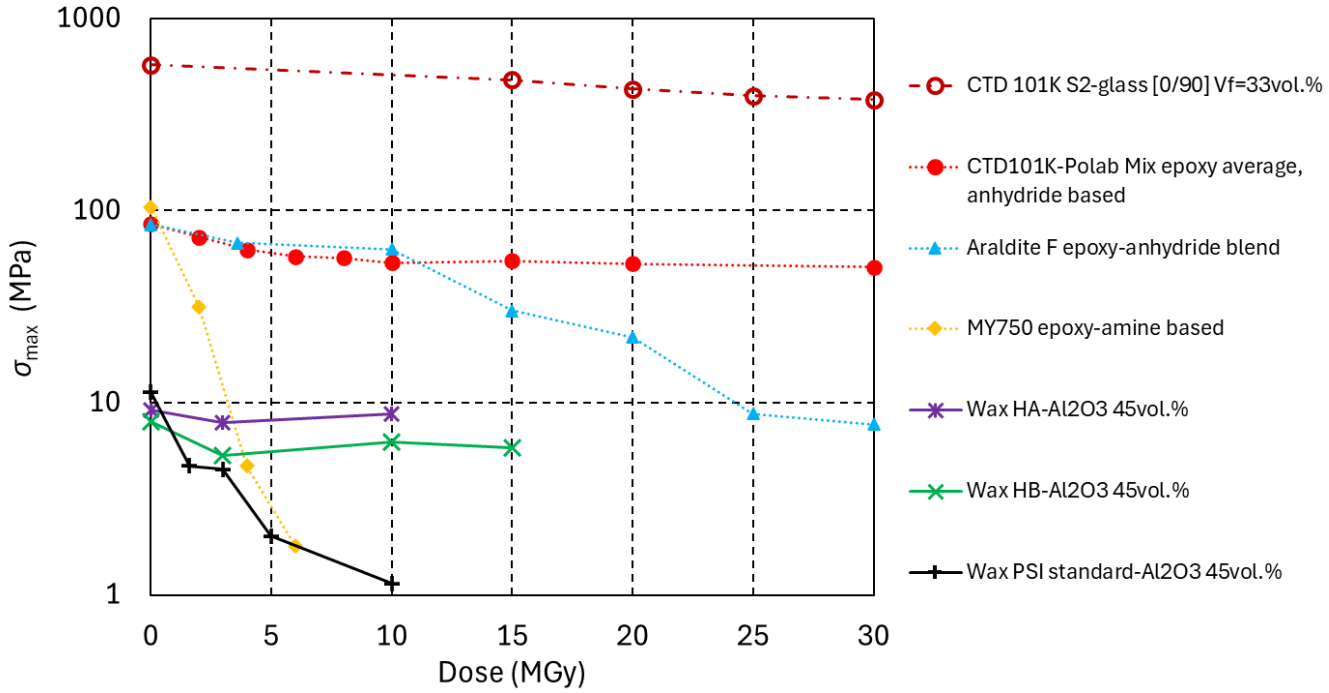
- For the Al_2O_3 filled wax types radiation damage can be monitored by measuring the stress at fracture (σ_{max}) in 3-point bending tests.
- After 5 MGy the PSI standard wax σ_{max} is reduced by about 80%.
- HA and HB wax σ_{max} changes only slightly up to 15MGy.



*3-point bending stress at fracture of different wax mixtures (*nominal Al_2O_3 content of 45 vol.%) *as a function of dose.* ***Courtesy Filip Louka.***

What is the effect of irradiation on the maximum wax loading stress in a coil?

- Different impregnation systems exhibit large σ_{max} differences. Wax fractures at low bending (tensile) stress.
- However, the main load case in accelerator magnet coils is transverse compressive loading.
- Under compression in a confined coil impregnation systems, wax loading stresses can be well above 100 MPa, assuming iso-stress in the different coil constituent's [6],[7].
- Under such conditions the effect of radiation on the maximum compressive loading stress is small.



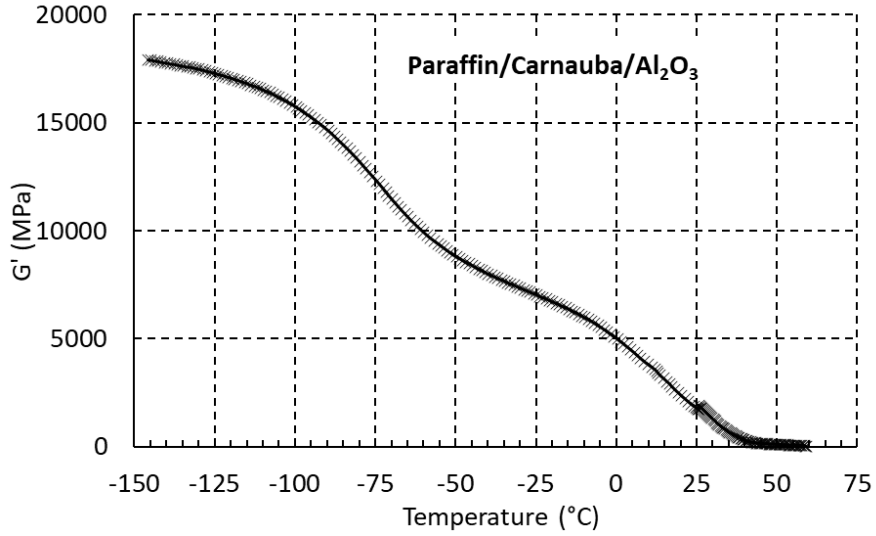
3-point bending stress at fracture of pure epoxy resins, epoxy composite and Al_2O_3 filled wax.

[6] F. Wolf, F. Lackner, M. Hofmann, C. Scheuerlein, D. Schoerling, D. Tommasini, "Effect of epoxy volume fraction on the stiffness of Nb3Sn Rutherford cable stacks" IEEE Trans. Appl. Supercond., 29(5), (2019), Art. no. 8401006

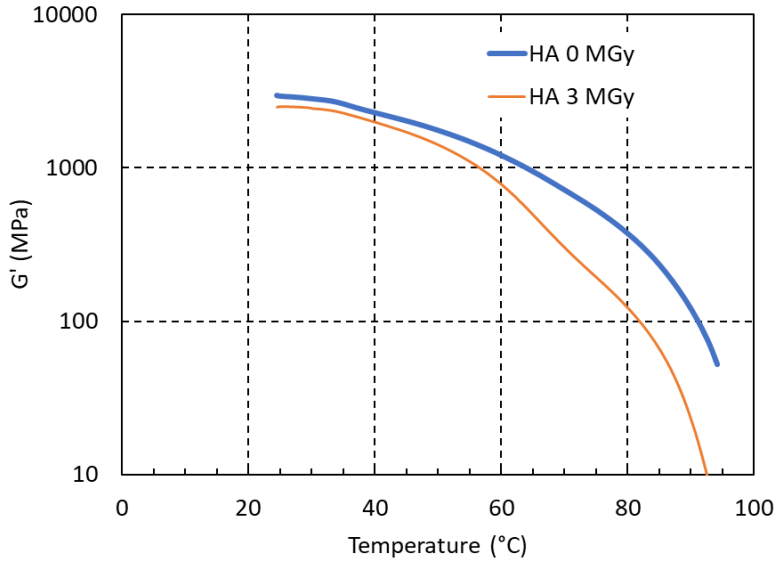
[7] C. Scheuerlein, F. Wolf, M. Lorentzon, M. Hofmann, "Direct measurement of Nb3Sn filament loading strain and stress in accelerator magnet coil segments", Supercond. Sci. Technol., 32, (2019), 045011 (7pp)

What is the effect of irradiation on the wax elastic properties?

- Attempts to measure elastic properties of Al_2O_3 filled wax were done by Dynamic Mechanical Analysis.
- Strong temperature dependence of elastic properties of Al_2O_3 filled wax.
- Preliminary results don't show signs of a strong change of elastic properties of the HA wax mixture at ambient at low temperature up to at least 3 MGy.



G'(T) of Paraffin/Carnauba wax mixture with Al_2O_3 particles. ***Courtesy of D.M. Parragh.***



G'(T) of HA wax mixture before and after 3 MGy irradiation.

Conclusion and outlook

- After 3 MGy the 3-point bending strength of the PSI standard wax mixture is reduced by about 50%.
- The “HA” high temperature melting point synthetic wax dose limit is in the order of 10 MGy. A dose of 10 MGy causes:
  - about 20% reduction of 3-point bending strength
  - about 8 °C reduction of melting temperature
  - about 50% reduction of melting enthalpy
- Under compressive loading in a confined coil impregnation systems, maximum loading stresses of the Al_2O_3 filled waxes are not strongly affected by doses up to at least 10 MGy.
- Irradiations in the absence of oxygen and at cold might cause less radiation damage. Irradiations at <20 K of few selected wax samples are planned.
- For application in superconducting accelerator magnets irradiation induced outgassing of wax needs to be considered.
- Irradiation testing of wax impregnation in a compression box sample could be considered as a verification test of an entire impregnation system.